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***J. J. Kruzic and R. O. Ritchie***

Materials Sciences Division, Lawrence Berkeley National Laboratory  
and  
Department of Materials Science and Engineering,  
University of California, Berkeley, CA 94720, USA

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# AN EXPERIMENTAL ASSESSMENT OF THE USE OF CRACK-OPENING DISPLACEMENTS TO DETERMINE INDENTATION TOUGHNESS FROM VICKERS INDENTS

**J. J. Kruzic and R. O. Ritchie\***

Materials Sciences Division  
Lawrence Berkeley National Laboratory, and  
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## ABSTRACT

Recently, a method for determining indentation fracture toughness from Vickers hardness indentations has been proposed which is based on a comparison of measured crack-opening displacements to computed values. To provide a first test of this method, experiments were conducted on a commercial silicon carbide ceramic, Hexaloy SA. Using the method, a toughness value within 10% of that measured using conventional fracture toughness testing was obtained; however, there was poor agreement between the measured and computed crack-opening profiles. Such discrepancies raise concerns about the suitability of this new method for determining the toughness of ceramics. Possible causes for these discrepancies are discussed, including subsurface lateral cracking and crack formation during loading.

## INTRODUCTION

Indentation toughness testing is an attractive alternative to more costly fracture mechanics experiments for determining the fracture properties of brittle materials. The typical method used involves measuring the radial cracks emanating from Vickers diamond microhardness indents and applying the semi-empirical relationship:<sup>1</sup>

$$K_c = \chi \sqrt{\frac{E}{H}} \frac{P}{a^{\frac{3}{2}}}, \quad (1)$$

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\* Corresponding author. Tel: +1-510-486-5798; fax: +1-510-486-4881. *E-mail address:* [roritchie@lbl.gov](mailto:roritchie@lbl.gov) (R. O. Ritchie)

where  $P$  is the indentation load,  $E$  is Young's modulus,  $H$  is the Vickers hardness,  $a$  is the radial crack length measured from the center of the indent, and  $\chi$  is an empirically determined "calibration" constant taken to be  $0.016 \pm 0.004$ .<sup>1</sup> One drawback of this method is that there is a large uncertainty ( $\pm 25\%$ ) in  $\chi$ ; consequently, the ability to obtain a precise value for the toughness is limited. Also in ceramics the value of  $H$  is not always invariant with respect to changing loads owing to indentation size effects, which gives further uncertainty to the  $K_c$  values obtained by this method (Eq. 1).<sup>2</sup> Finally, for materials that exhibit rising R-curve behavior due to crack bridging or transformation toughening, additional uncertainties arise due to the fact that indentation toughness essentially gives a random point on the R-curve.

A new, non-empirical, method utilizing newly available solutions for the crack-opening displacements of Vickers indentation cracks has recently been proposed by Fett for determining the indentation toughness.<sup>3</sup> With this method, the crack-opening profile,  $u(r)$ , for a linear-elastic Vickers indent crack is expressed in terms of the near-tip stress intensity,  $K_{tip}$ , such that:<sup>3</sup>

$$u(r) = \frac{4K_{tip}\sqrt{b}}{\pi E'} \left( A\sqrt{1 - \frac{r}{a}} + B\left(1 - \frac{r}{a}\right)^{\frac{3}{2}} + C\left(1 - \frac{r}{a}\right)^{\frac{5}{2}} \right), \quad (2)$$

where  $E'$  is the plane strain modulus (i.e.,  $E' = E/(1-\nu^2)$ , where  $\nu$  is Poisson's ratio) while  $r$ ,  $b$ , and  $a$  are the radial position, contact-zone radius, and the crack length, respectively, as measured from the center of the indent. The coefficients  $A$ ,  $B$ , and  $C$  are given by:<sup>3</sup>

$$A = \sqrt{\frac{\pi a}{2b}}, \quad (3)$$

$$B \cong 0.011 + 1.8197 \ln\left(\frac{a}{b}\right), \quad (4)$$

$$C \cong -0.6513 + 2.121 \ln\left(\frac{a}{b}\right). \quad (5)$$

Simplifying the first term in Eq. 2 gives the Irwin near-tip crack opening solution:

$$u(r) = \frac{K_{tip}}{E'} \sqrt{\frac{8(a-r)}{\pi}}. \quad (6)$$

For the simple case of an ideally brittle, non-toughened ceramic, Eqs. 2-5 provide a means for deducing the intrinsic (crack initiation) toughness,  $K_o$ , from the measured crack-opening profile. One necessary assumption, as in other indentation toughness methods, is that cracking occurs due to the residual tensile stresses during unloading and accordingly crack arrest occurs when the near-tip stress intensity  $K_{tip} = K_o$ .

This method also has potential for use with bridging ceramics, such as grain-elongated  $\text{Si}_3\text{N}_4$  and ABC-SiC.<sup>4-6</sup> By separating out the bridging displacements, which distort the crack-opening profile, from that expected in the traction-free

case (i.e., that given by Eq. 2-5)<sup>3</sup>, the intrinsic toughness, or crack-initiation point on the R-curve, may be determined, as well as values for the bridging contribution to the toughness.

With this goal in mind, the present objective is to assess the suitability of Fett's method for assessing fracture toughness.<sup>3</sup> A commercial silicon carbide, Hexoloy SA, was chosen for this study for several reasons. Firstly, Hexoloy SA fractures transgranularly without crack bridging. Consequently, there is a single-valued fracture toughness for this material, which is independent of crack length. i.e., no R-curve behavior; moreover, the toughness of this material has been well characterized.<sup>4,6</sup> Additionally, a well defined radial crack system, necessary for this study, can be readily produced by indenting silicon carbide ceramics.<sup>1,7</sup> Finally, as silicon carbides can be readily produced to develop grain bridging,<sup>4,6</sup> the study can be extended to the same nominal material only with R-curve behavior. In order to test the validity of this new method, results are compared to those obtained by both conventional fracture mechanics testing, using precracked specimens, and conventional indentation toughness methods, using Eq. 1.

## EXPERIMENTAL PROCEDURES

Disks of a pressureless sintered silicon carbide, Hexoloy SA, were ground flat and lapped to a 1  $\mu\text{m}$  finish using diamond compounds. A 4 kg load was used to produce Vickers indentations. While using a range of loads would be ideal for this assessment, lower loads produced too short of indent cracks while larger loads caused surface chipping. Accordingly, a 4 kg indentation load allowed the maximum radial crack lengths without chipping the sample surface. Three methods for determining indentation toughness were compared:

1. *Crack-opening profile (COP) method:* Using the proposed method of Fett,<sup>3</sup> denoted here as the COP method, the intrinsic toughness,  $K_{\text{I}}$ , was determined by measuring crack-opening profiles,  $u(r)$ , and computing the value of  $K_{\text{I}}$  using Eqs. 2-5. Crack openings were measured in a field-emission scanning electron microscope (FESEM), with a maximum obtained resolution of 5 nm for the full crack opening,  $2u$ . The optimal value of  $K_{\text{I}}$  was determined to be that which produced the best least-squares fit of Eqs. 2-5 to the experimentally measured crack-opening profile.
2. *Near-tip (NT) method:* To assess whether the full crack-opening profile was needed for this method, a identical technique to the COP method was utilized *except* that only the near-tip crack-opening data were used. Accordingly, for this near-tip (NT) method, the Irwin solution (Eq. 6) was used instead of Eqs. 2-5; this method was carried out using 5, 10, 15, and 20  $\mu\text{m}$  of crack-opening data.
3. *Traditional indentation toughness (TIT) method:* Additionally, the above results were compared to toughness value obtained using the standard indentation toughness procedures.<sup>1</sup> Specifically, the indent size and crack length were measured and the toughness computed using Eq. 1.

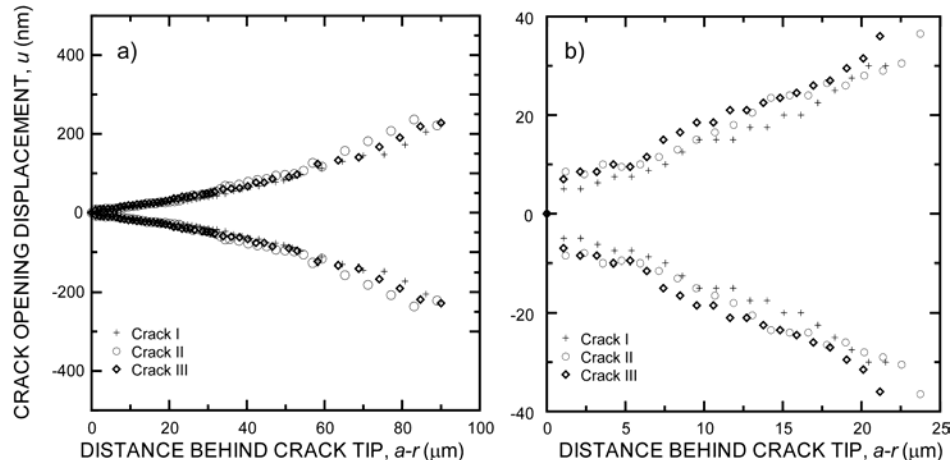


Fig. 1: Crack-opening profiles for a) the entire indent cracks and b) the near-tip regions.

Finally, all indentation toughness results were compared to values measured using *conventional fracture mechanics methods*, based on studies on precracked disk-shaped compact-tension specimens.<sup>5,7</sup>

## RESULTS

Fig. 1a shows the full crack-opening profiles measured for three Vickers indent cracks, while Fig. 1b shows a close up of the near-tip region for each crack. The three cracks are denoted I, II, and III. Fig. 2 shows micrographs of cracks I and II along with a cross section that reveals the nature of the subsurface cracking which occurs in this material. Estimates of the intrinsic toughness for Hexoloy SA,  $K_{0i}$ , were made using the results from Fig. 1 along with Eqs. 2-5, i.e., using the COP method. The deduced  $K_{0i}$  values were 2.0 MPa  $\sqrt{\text{m}}$  for crack I and 2.3

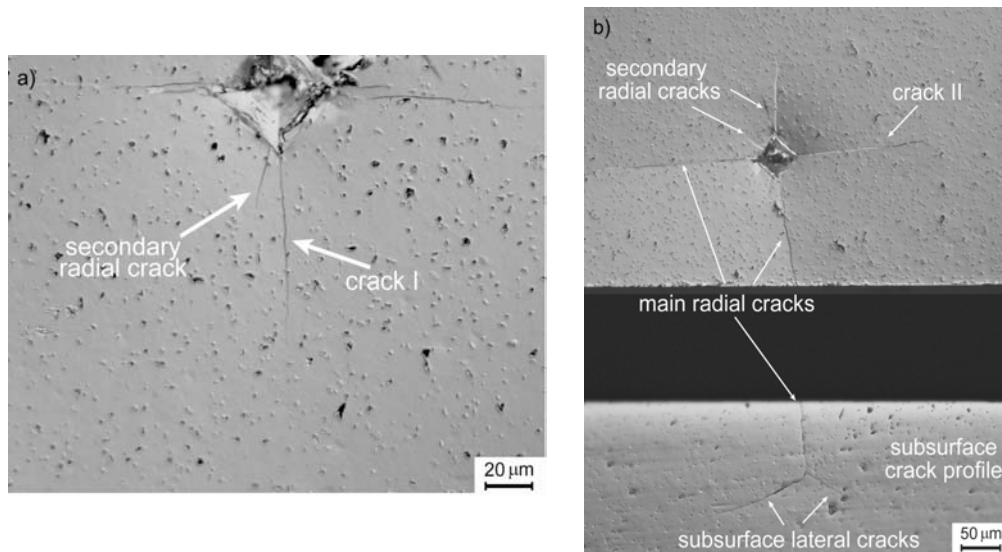


Fig. 2: Micrograph showing a) crack I and b) crack II along with a subsurface crack profile

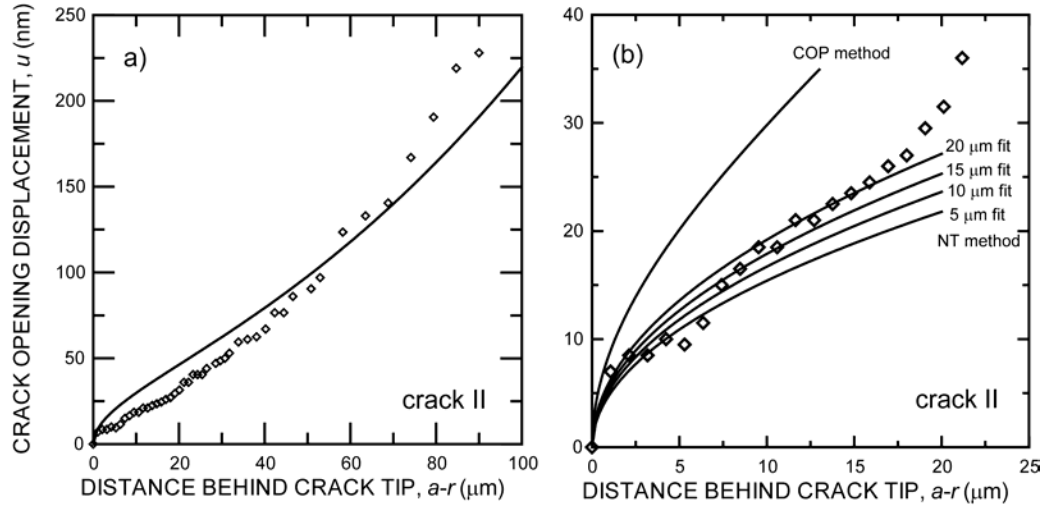


Fig. 3: Part a) shows the computed crack opening for crack II based on the COP method along with the full measured crack-opening profile while b) shows the near-tip region along with the computed openings based on the NT and COP methods.

MPa  $\sqrt{\text{m}}$  for cracks II and III. Using these deduced toughness values, the crack-opening profiles were computed for each crack and compared to the measured crack openings. The results looked similar for each case, with Fig. 3a showing the predicted crack opening for crack II using  $K_0 = 2.3 \text{ MPa } \sqrt{\text{m}}$ . The above results obtained using the COP method are tabulated in Table I along with the results from the NT method. The later results were obtained using only the near-tip data (Fig. 1b) with the Irwin crack-opening solution (Eq. 6), and were found to be dependent on the amount of data used for the fit (Table I). As for the COP method, the best fit near-tip crack profiles were computed for each case. Again these results were similar for each crack; one example set of results, for crack II, are shown in Fig. 3b.

Table I: Fracture toughness results using crack-opening profile measurements (COP and NT methods)

	Crack I	Crack II	Crack III
COP method (Eqs. 2-5)	2.0 MPa $\sqrt{\text{m}}$	2.3 MPa $\sqrt{\text{m}}$	2.3 MPa $\sqrt{\text{m}}$
NT method (Eq. 6) 5 $\mu\text{m}$ fit	0.93 MPa $\sqrt{\text{m}}$	1.3 MPa $\sqrt{\text{m}}$	1.4 MPa $\sqrt{\text{m}}$
NT method (Eq. 6) 10 $\mu\text{m}$ fit	1.0 MPa $\sqrt{\text{m}}$	1.4 MPa $\sqrt{\text{m}}$	1.2 MPa $\sqrt{\text{m}}$
NT method (Eq. 6) 15 $\mu\text{m}$ fit	1.2 MPa $\sqrt{\text{m}}$	1.5 MPa $\sqrt{\text{m}}$	1.4 MPa $\sqrt{\text{m}}$
NT method (Eq. 6) 20 $\mu\text{m}$ fit	1.3 MPa $\sqrt{\text{m}}$	1.6 MPa $\sqrt{\text{m}}$	1.5 MPa $\sqrt{\text{m}}$

Finally, to compare the indentation toughness results obtained from the crack-opening profile measurements to those obtained by the traditional indentation toughness method,<sup>1</sup> measurements were made for cracks at four indents, giving an average indentation toughness (TIT method) of  $2.1 \pm 0.7 \text{ MPa}\sqrt{\text{m}}$  for Hexoloy SA.

## DISCUSSION

The results obtained in the present study using the COP method compare favorably with the typical reported values of roughly  $2.5 \text{ MPa}\sqrt{\text{m}}$  obtained using conventional fracture mechanics testing with disk-shaped compact-tension specimens.<sup>4,6</sup> Specifically, for cracks II and III, the deduced value of  $2.3 \text{ MPa}\sqrt{\text{m}}$  falls within 10% of typical reported values. For crack I, a slightly lower value ( $2.0 \text{ MPa}\sqrt{\text{m}}$ ) was obtained; however, Fig. 2a reveals that there was a significant secondary radial crack near the main crack I. Such secondary cracking may have relieved some of the residual stress which holds the crack open, thus affecting the crack-opening profile and the deduced toughness values for crack I. Cracks II and III were specifically chosen such that no (crack II) or minimal (crack III) secondary cracking could be observed near the main crack. Thus, considering only the results for cracks II and III, it may be concluded that reasonable estimates of the intrinsic toughness of Hexoloy SA may be obtained using the COP method if the cracks analyzed are specially chosen such that secondary radial cracking is at a minimum.

One major disadvantage of the semi-empirical traditional indentation toughness (TIT) method is the large scatter in the results; indeed, in the present study a wide range of values from 1.4 to  $2.8 \text{ MPa}\sqrt{\text{m}}$  was obtained for Hexoloy SA. Although this range overlaps with the typical reported values of  $\sim 2.5 \text{ MPa}\sqrt{\text{m}}$  obtained from conventional fracture mechanics methods,<sup>4,6</sup> the lowest values of the range differ by some 45%. Conversely, the COP method produced values within 10% of the expected value. Furthermore, the deduced toughness value,  $K_{\text{IC}}$ , was still within 20% for the case of crack I, where secondary radial cracking is believed to have affected the results. Thus, these first results indicate there is definite promise of using crack-opening displacement measurements to deduce toughness values for ceramics.

There are serious concerns about using this method, however, based on the large discrepancies between the computed and measured crack-opening profiles seen in Fig. 3. The results in Fig. 3 were typical for all three cracks in that the crack-opening shape did not agree with that predicted by Eqs. 2-5, particularly in the near-tip region. Toughness results obtained using the NT method further illustrate this point. Fitting the Irwin solution (Eq. 6) to the near-tip crack-opening data yielded toughness values up to 50% lower than that obtained using the COP method. Although the COP method takes the entire crack opening into account, and accordingly should give more accurate results, such large discrepancies were not expected. These results indicate that the actual near-tip crack openings are much smaller than would be expected for a crack held open

just below the condition for critical fracture, i.e.,  $K_{\text{tip}} = K_{\text{o}}$ . It is important to note that Hexoloy SA fractures transgranularly, so the smaller than expected crack openings may not be attributed to crack-face interactions such as bridging.

The observed discrepancies may be attributed to several possible factors. The first of these is the presence of subsurface lateral cracking, as shown in Fig. 2b. While subsurface lateral cracks are not uncommon during Vickers indentation,<sup>8-10</sup> the effects of such cracking have largely been ignored in the analysis methods for indentation toughness. For the traditional indentation toughness (TIT) method, lateral cracking is not considered in current analyses, i.e., Eq. 1;<sup>1</sup> however, it is likely to account for some of the uncertainty in the empirical calibration factor. One effect of lateral cracking is to alter the residual stress field, which in turn affects the crack-opening profiles and hence the toughness values deduced by the COP and NT methods. Specifically, relaxation of residual stresses would permit the cracks to close more than expected or have a different opening shape than expected; indeed, both of these scenarios are observed in the results of Fig. 3. Accordingly, it is expected that future developments in this method will need to account for such lateral cracking when predicting crack-opening shapes.

Finally, the basic assumption of all current indentation toughness methods, that cracking occurs during unloading as a result of the residual tensile stresses, has been brought into question by the work of Cook and Pharr.<sup>9</sup> Specifically, their *in situ* optical microscopy studies of transparent materials showed that while some glasses exhibit the expected behavior of cracking during unloading, several transparent ceramic materials in fact exhibited cracking immediately upon loading the sample. Such results raise questions about the general applicability of all indentation toughness methods, including the TIT method which has now been used for more than twenty years. It is currently unknown if cracking occurs upon loading or unloading in SiC; however, the possibility of cracking during the loading portion of indentation may contribute to the observed discrepancies between predicted and measured crack openings seen in Fig. 3.

In summary, it is clear that there are significant uncertainties underlying a general indentation toughness test method for ceramics based on the measurement of crack-opening displacements. While the method as applied here may work well for some ceramic materials, further experiments for a range of brittle materials are needed. Furthermore, for silicon carbide, it is apparent that a more thorough understanding of the mechanisms and interactions of Vickers indent cracking is needed before such a method may be suitably applied for reliable toughness determination. Thus, while the crack-opening profile (COP) indentation toughness method proposed by Fett<sup>3</sup> indeed holds promise, there is a need for further investigation in this area.

## CONCLUSIONS

Based on an experimental study to assess the use of crack-opening displacements to determine the fracture toughness of a commercial silicon carbide



ceramic, Hexoloy SA, from Vickers indentation cracks, the following conclusions are made:

1. An intrinsic toughness value of  $K_{IC} = 2.3 \text{ MPa}\sqrt{\text{m}}$ , which was within 10% of values reported using standard fracture mechanics specimens, was obtained by fitting the expected full crack-opening profile to the measured openings. Such close correspondence illustrates the potential of such methods.
2. The deduced toughness was lower ( $2.0 \text{ MPa}\sqrt{\text{m}}$ ) for an indentation crack where significant secondary radial cracking was evident. In this case, secondary radial cracking was believed to affect the results by relieving some of the residual stresses, resulting in smaller crack openings and a smaller deduced toughness value.
3. In all cases, there were significant discrepancies between the measured and computed crack openings, even in the absence of secondary radial cracking. Possible explanations for these discrepancies include subsurface lateral cracking, which was directly observed and can affect the residual stress field, and the possibility of cracking during the loading portion of the indentation process.
4. Although it appears that methods using the crack-opening displacements to determine the fracture toughness of ceramics from Vickers indents hold promise, a more thorough understanding of the complexities of indent cracking is needed for this to become a reliable and accurate test method.

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